National Defence
Research and Development Branch

Défense nationale
Bureau de recherche et développement

DREA Contractor Report 98/441

# ULTRAVIOLET SPECTROSCOPY TOTAL OIL CONTENT OF WATER

by

Clive MacGregor — Valerie Gray

SEATECH LIMITED
958 Barrington Street
Halifax, Nova Scotia, Canada
B3H 2P7

## CONTRACTOR REPORT

Prepared for

Defence Research Establishment Atlantic

Centre de Recherches pour la Défense Atlantique

Canada

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Accepted by : _____
D.E. Veinot - Contract Scientific Authority

W7707-7-5038/001-HAL
Contract Number

October 1998

CONTRACTOR REPORT

Defence Research Establishment Atlantic

Centre de Recherches pour la Défense Atlantique

Canada

## Abstract

Defence Research Establishment Atlantic contracted Seatech Ltd. To assess the capability of UV spectrophotometry to detect oil in water as may be discharged from DND vessels.

For the study a brief literature review was undertaken and five oils (Diesel Fuel, Naval Distillate, Diesel Motor Oil, Synthetic Lubricating Oil, Hydraulic Fluid) assessed over the range 5 to 100 ppm in water. In addition some "real" samples from a naval vessel at Halifax were tested by UV spectrophotometry and standard methods for oil content. The findings were as follows:

1. Both UV absorbance and fluorescence will detect oil in water in dissolved and particulate phases at concentrations below 10 ppm, with a detection limit of approximately 1 ppm.
2. The portion of oil dissolved, compared to the total oil in water varied from 25% to 75%, with greater dissolution for fuel oils and lesser for lubes/hydraulics.
3. Wavelength of maximum UV absorbance or fluorescence, and response per unit concentration varied with oil type: thus making the choice of a standard for calibration of an oil detection monitor difficult.

It was determined that response versus oil in water concentration, linearity, and minimum level of detection were better for UV fluorescence than for UV absorbance. The literature review indicated UV fluorescence was generally preferred over UV absorbance and other detectors, and was less subject to interferences from bilge chemicals or solids compared to UV absorbance. "Real" samples were found to contain very high levels of oil in water and UV spectrometry did not accurately estimate these levels of oil. UV spectrophotometry, particularly UV fluorescence, was most accurate at 25 ppm or less oil in water.



## Résumé

Le Centre de recherches pour la défense (Atlantique) a passé un contrat avec la Seatech Ltd. en vue d'évaluer la possibilité de détecter par spectrophotométrie UV la présence dans l'eau d'hydrocarbures des types susceptibles d'être rejetés par les navires du MDN.

Pour réaliser ces travaux, une brève étude bibliographique a été effectuée et cinq types d'hydrocarbures (carburant diesel, distillat pour la marine, huile pour moteur diesel, huile lubrifiante synthétique et liquide hydraulique) ont été évalués à des concentrations allant de 5 à 100 ppm dans l'eau. De plus, la teneur en hydrocarbures de quelques échantillons « véritables », provenant d'un navire de guerre, à Halifax, a été déterminée par spectrophotométrie UV et grâce à des méthodes normalisées. Voici les résultats obtenus :

1. L'absorption et la fluorescence UV permettent de détecter les hydrocarbures dissous et en suspension dans l'eau en concentrations inférieures à 10 ppm, avec une limite de détection d'environ 1 ppm.
2. La quantité d'hydrocarbures dissous représente de 25 à 75 % de la quantité totale d'hydrocarbures dans l'eau, la quantité de carburant dissous étant plus grande que la quantité d'huiles lubrifiantes et de liquides hydrauliques en solution.
3. La longueur d'onde d'absorption ou de fluorescence UV maximale et la résponse par unité de concentration varient selon le type d'hydrocarbure. Il est donc difficile de choisir un étalon pour l'étalonnage d'un détecteur d'hydrocarbures.

La fluorescence UV permettait d'obtenir une meilleure réponse en fonction de la concentration d'hydrocarbures dans l'eau, une meilleure linéarité et un meilleure limite de détection que l'absorption UV. L'étude bibliographique a permis d'établir que la fluorescence UV était, en général, préférée à l'absorption UV et à d'autres méthodes de détection, car elle est moins sujette aux perturbations causées par les produits chimiques ou les solides de fond de cale. Les échantillons « véritables » présentaient des concentrations très élevées d'hydrocarbures dans l'eau, que la spectrophotométrie UV ne permettait pas de déterminer avec précision. La précision obtenue par spectrométrie UV, en particulier par fluorescence UV, est très élevée à une concentration égale ou inférieure à 25 ppm d'hydrocarbures dans l'eau.

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## Executive Summary

Defence Research Establishment Atlantic contracted Seatech Ltd. to assess the capability of UV spectrophotometry to detect oil in water as may be discharged from DND vessels. The project addressed two prime questions: Is UV spectrophotometry (absorbance and fluorescence) a technically viable manner by which to detect oil in discharge water at less than 10 ppm, and is oil at low levels in discharge waters dissolved or particulate?

For the study a brief literature review was undertaken and five oils (Diesel Fuel, Naval Distillate, Diesel Motor Oil, Synthetic Lubricating Oil, Hydraulic Fluid) assessed over the range 5 to 100 ppm in water. In addition some "real" samples from a naval vessel at Halifax were tested by UV spectrophotometry and standard methods for oil content.
The findings were as follows:

1. Both UV absorbance and fluorescence will detect oil in water in dissolved and particulate phases at concentrations below 10 ppm, with a detection limit of approximately 1 ppm.
2. The portion of oil dissolved, compared to the total oil in water varied from 25 to 75, with greater dissolution for fuel oils and lesser for lubes/hydraulics.
3. Wavelength of maximum UV absorbance or fluorescence, and response per unit concentration varied with oil type; thus making the choice of a standard for calibration of an oil detection monitor difficult.

It was determined that response versus oil in water concentration, linearity, and minimum level of detection were better for UV fluorescence than for UV absorbance. The literature review indicated UV fluorescence was generally preferred over UV absorbance and other detectors, and was less subject to interferences from bilge chemicals or solids compared to UV absorbance. "Real" samples were found to contain very high levels of oil in water and UV spectrometry did not accurately estimate these levels of oil. UV spectrophotometry, particularly UV fluorescence, was most accurate at 25 ppm or less oil in water.

It is recommended that additional study be undertaken on how to calibrate any UV oil detection monitor, including choice of oil mixture, wavelength and procedure.

## 1.0 Introduction

Department of National Defence, Defence Research Establishment Atlantic, Halifax, N.S. had a requirement to investigate and evaluate the use of ultraviolet (UV) spectroscopy for the determination of total (particulate and dissolved) oil in water. The work outlined here compared measurements of total oil in water by both UV absorbance and UV fluorescence, after solvent extraction and on direct measurements of aqueous solutions. In total, 1000 plus analyses (UV absorbance, UV fluorescence, GC/FID and GC/MSD) were undertaken to assess the viability of using UV Spectroscopy for detection of the oil content of water, as expected to be discharged from Canadian Naval vessels after treatment.

Oils discharged from seawater ballasted fuel tanks or other Naval operations will contain both dissolved and particulate oil phases. Discharge of oil is presently regulated to 15 ppm or less for both phases, but for many operations the regulated level may be much lower. The detection of dissolved oil in situ was assessed for Canadian Naval applications, particularly for seawater ballasted fuel tanks on CPF vessels. The major concern was the inability of present oil detection monitors to detect accurately both dissolved oil and particulate oil phases of various oil types (Diesel Motor Oil, Diesel Fuel, Hydraulic Fluid, Naval Distillate, Synthetic Lube Oil) in the 0 to 100 ppm range. This study addressed the generic ability of ultraviolet spectroscopy techniques (UV absorbance and UV fluorescence) to detect both dissolved and particulate oil in the range of interest.

## 2.0 Methods

### 2.1 Sample Preparation

The sample jars used for the oil in water tests were 500 mL mason jars with tin-foil lined caps. These jars were pre-cleaned (hexane rinsed) and allowed to dry a minimum of 24 hours. 450 mL of freshwater (distilled oil free water) or seawater (prepared with artificial sea salt and distilled oil free water) were added to the jars as needed. Seawater was prepared by adding approximately 400 grams of *Forty Fathoms* artificial sea salt to 10 L of distilled water and adjusting until a salinity of 30-33 ppt was achieved. Salinity was measured using a YSI-33 salinity meter.

The oils selected for testing were: two Distillate Fuels; Naval Distillate and Diesel Fuel, and three lubes/hydraulic oils; Diesel Motor Oil, Synthetic Lube and Hydraulic Oil. For oil spiking of the test water it was necessary to make highly concentrated stock standards (approximately 50% oil in hexane) to spike the water at low levels of oil (5 - 100 mg/L). Once the water was spiked it was placed on a Gyrotory Shaking Apparatus (Model G33) at 200 rpm for one hour. The "total portion" was sub-sampled immediately after being removed from the shaker by inserting a 15 mL volumetric pipette half way into the jar and extracting the sample. The bottle was allowed to remain undisturbed for another hour and then the "dissolved portion" was extracted in the same manner.

Once the samples were removed, the 15 mL portions to be measured on the water directly were placed in a 30 mL disposable glass vials with a foil lined cap and placed in a dark box until they could be measured. The 15 mL samples to be extracted were placed in 30 mL glass vials and had 15 mL of Methylene chloride added, placed on the Gyrotory Shaker at 200 rpm for 1 hour. After 1 hour of shaking the samples were removed and placed in a dark box until they could be measured. Measurements were always conducted as soon as possible after sample preparation.

### 2.2 Laboratory Equipment

#### 2.2.1 Spectrophotometry

Measurements were conducted using three different spectrophotometers. A Shimadzu

synchronous scanning fluorescence spectrophotometer (Model RF-5301 PC) was used for determining fluorescence spectra and measurements at fixed wavelengths. A Hewlett Packard Diode Array Scanning Spectrophotometer (Model HP8452A) was used for determining UV absorbance spectra. After an appropriate absorbance wavelength had been determined for all five oils, a Shimadzu UV spectrophotometer (Model UV-120-02) was used for measurements at the fixed wavelength. All measurements were conducted using a 1 cm quartz cuvett. All instruments were zero adjusted with either methylene chloride or seawater as required.

For UV absorbance oil in water (direct or extracted) quantification at the fixed wavelength 235 nm was chosen. For fluorescence quantification excitation was 254 nm (a strong mercury lamp light intensity wavelength), with emission measured at 380 nm. These wavelengths were chosen after discussion with the Scientific Authority, and represented absorbance or emission wavelengths that were considered the best compromise of different absorbance or emission wavelength maxima for all oils to be tested.

### 2.2.2 Gas Chromatography

Standards of the oils in methylene chloride, and oil in water as extracted at a one to one ratio water to solvent, were analyzed using a Hewlett Packard (HP) 5890 Gas Chromatograph with a Flame Ionization Detector (GC-FID) with a SPB-5 column (5% phenylmethyl silicone). These same samples were also analyzed using a HP 5890 Gas Chromatograph with a HP 5972 Mass Selective Detector (GC-MS). Data was acquired for both GC-FID and GC-MS analyses on HP Chem Station software. With this method it was possible to determine the relative abundance of aliphatic to aromatic components using GC-MS.

## 2.3 BTEX / TPH Analysis

Total Petroleum Hydrocarbons (TPH); Benzene, Toluene, Ethylbenzene and Xylenes (BTEX) in shipboard water samples were determined by cold solvent extraction into Methylene chloride followed by GC-FID. The method detection limits for Benzene, Toluene, Ethylbenzene and Xylenes are 0.005 mg/L each. The method detection limits for gasoline, diesel and lubricating oil are 0.1 mg/L each.

## 2.4 Literature Search

Several indexes were checked during the literature search part of the project. Through Environmental Abstracts the information "oil detection" monitors dealt with remote sensing of oil spills. Various search terms were used for each index. Through Compendex "Marine pollutant equipment", "shipboard wastes", "oil", "detection", and "monitor" were some of the key searches. Relevant information could not be found through the National Technical Information Server (NTIS). A search of US Government Publications and Chemical Abstracts for the last five years, failed to yield relevant information. Online searches through Dialogue's various databases were used with Petroleum Abstracts being the most useful.

## 2.5 Collection of Shipboard Samples

Five "real" water samples were collected from the HMCS Athabaskan. The samples were identified as follows:

1) AMR Bilge
2) IAC Plant Bilge
3) #5 Settling Tank
4) Aft Stripping Tank
5) Gland Space Bilge

The samples were collected in 1L amber glass bottles by Seatech personnel. The bottles were dipped into the appropriate bilge and allowed to fill to the top (no head-space). Tank samples (#3 and #4) were retrieved by bottom triggered Bacon Bomb sampler. Samples were then returned to the Seatech laboratory and refrigerated for future analysis.

These samples were analyzed by standard methods of cold solvent (methylene chloride) extraction and GC/FID analysis on an HP5890 GC. Concurrently the samples were tested for oil in water by direct reading on UV fluorescence (excitation at 254 nm, emissions at 380 nm) and UV absorbance (absorbance at 235 nm) as well as spectral scans.

## 3.0 Results

### 3.1 Literature

Literature was reviewed to establish the use of UV Spectroscopy in detection of oil in water for discharge monitoring, in total 12 references were retrieved. The majority (6) of this literature was by manufacturers promoting their specific monitors. References were found for the use of both UV Absorbance and UV Fluorescence in discharge oily water monitoring. However, UV spectroscopy has not been the historical monitor of choice. White light (droplet scattering) was found to be the most common detector followed by Infrared spectroscopy, but neither unit could detect dissolved oil <10 ppm. Only the UV monitors showed any capability to detect dissolved oil at levels below 5-10 ppm.

The most common UV spectroscopic technique was found to be fluorescence (three manufacturers), with UV absorbance being only noted for two smaller companies. Levels of detection for fluorescence were reported at 1 ppm and less, sufficient for the expected Canadian Naval requirement. Two papers noted below were the most relevant, being recent comparison of various oil in water monitoring techniques.

1) Hadfield, D; Tulloch, S; An Overview Of On-Line Oil-In-Water Monitor Testing At Orkney Water Test Centre, 3rd IBC Tech Serv Ltd Water Manage Offshore Int Conf (Dyce, Scot, 6-7 Oct 1993), proceeding 20pp, 1993
2) Henriquez, L, Intercalibration of Automatic Continuous Oil-In-Water Monitors With The Parcom Procedures, (source; same as above), 1993

In both of these documents UV fluorescence oil detection monitors received good ratings. Excluding the references noted above there was very little recent literature on oil detection monitors, and most of this was by manufacturers proving their unit was the best. Literature on oil in water monitors appeared to peak in the mid 1970's. The UV fluorescence unit was, for recent installations, the most common unit.

The literature described several common problems for oil in water monitors as noted below.

-solids interfering with readings (lesser effect in UV fluorescence)
-coating of lenses with oil or algae
-interferences by chemicals or cleaners (lesser effect UV fluorescence)

The literature did note some units only responded to particulate oil, not dissolved oil. Literature also indicated that at low levels of oil in water (~25 ppm or less) significant portions of the oil, up to 75% or greater were present in the dissolved form.

### 3.2 UV Absorbance and Fluorescence Spectra of Oil Standards In Solvent

These spectra showed that fuel oils and lubricating/hydraulic oils had different maximum UV absorbance and fluorescence wavelengths. The UV absorbance peaked at 235 nm for fuel oils and at 280 nm for lube/hydraulic oils (Figure 3.2.1). In fluorescence, the fuel oils had maximum fluorescence at 315 nm emission. (excitation 254 nm) and a broad band response for lube / hydraulic oils over the range 350-410 nm emission (Figure 3.2.2). As expected, response per unit concentration (either UV absorbance or fluorescence) was different for each oil, with fuel oils showing the highest response and hydraulic oils the lowest response per unit concentration (Figures 3.2.3 & 3.2.4).

### 3.3 UV Absorbance And Fluorescence Determination of Oil Standards In Water

These spectra were very similar though of slightly less intensity to spectra observed in solvent (methylene chloride) for both total and dissolved portions of the five oils. Using absorbance, the percent dissolved for the oils at 25 ppm oil in water ranged from 0% for Hydraulic Fluid to 80% for Diesel Fuel (Table 3.3.1). Using fluorescence, the percent dissolved for the oils at 25 ppm oil in water ranged from 0% for Diesel Motor Oil to 83% for Synthetic Lube Oil (Table 3.3.2). There was considerable variability of dissolution between replicates for both modes of measurements.

Both aromatics and aliphatics were found in the dissolved portion of oil in water, as determined by GC/MS. GC/FID chromatograms of dissolved portions of Naval Distillate and Diesel Motor Oil are presented in Figures 3.4.1 and 3.4.2.. In both these examples the dissolved and total chromatograms are similar, though of different intensity.

### 3.4 Time For Maximum Dissolution of Oils In Seawater

The time for maximum dissolution on oil in seawater was determined by trials of the various oil types spiked into water at a concentration of 20 mg/L. The "dissolved portions" were assessed

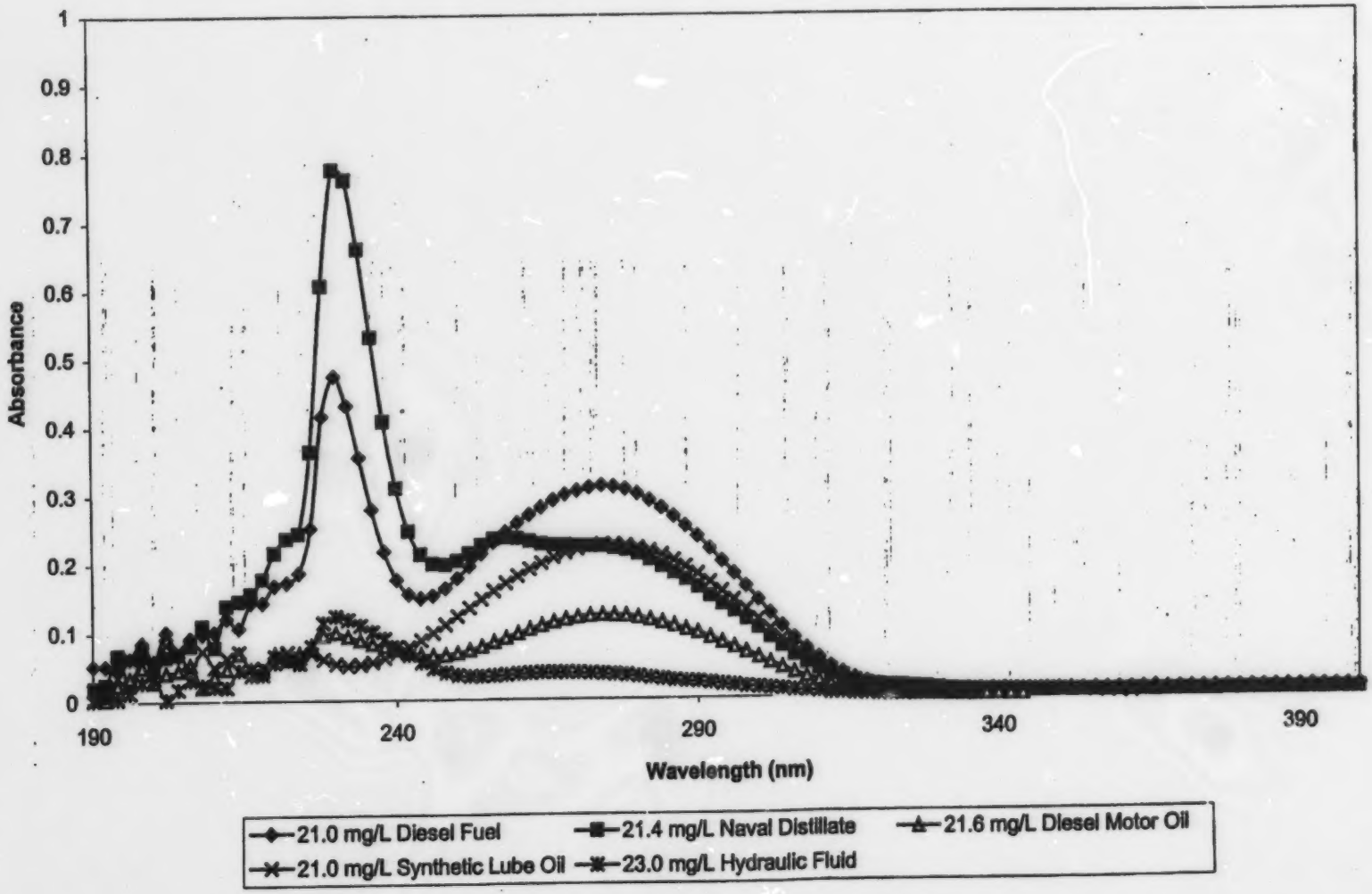
U.V. Absorbance Spectra 25 ppm Oil Standards in CH2Cl2
Absorbance
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
190
240
290
340
390
Wavelength (nm)
21.0 mg/L Diesel Fuel
21.4 mg/L Naval Distillate
21.6 mg/L Diesel Motor Oil
21.0 mg/L Synthetic Lube Oil
23.0 mg/L Hydraulic Fluid

**Figure 3.2.1**

## Emission Spectra of Oil Standards in $CH_2Cl_2$ (excitation at 254 nm)

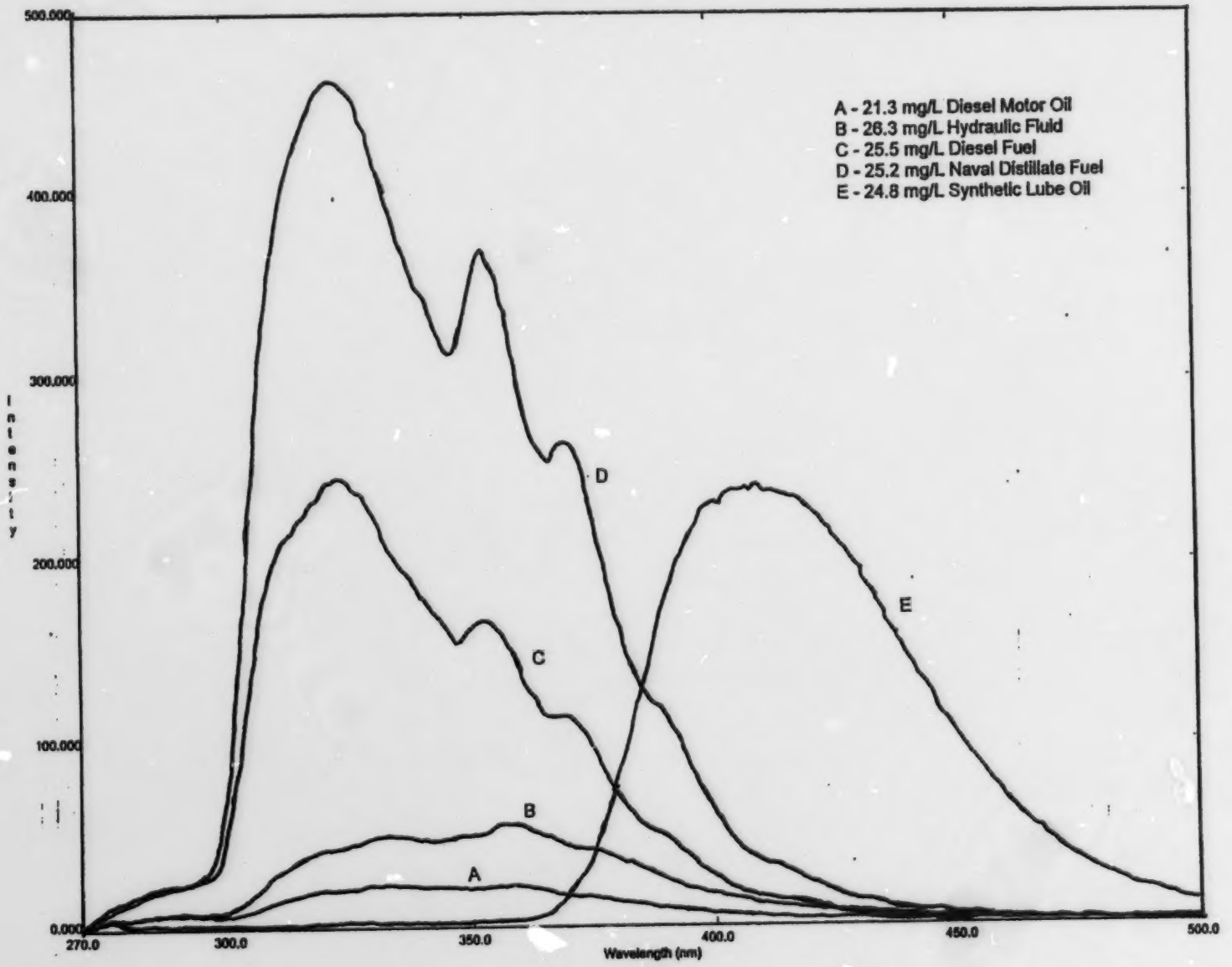


Figure 3.2.2

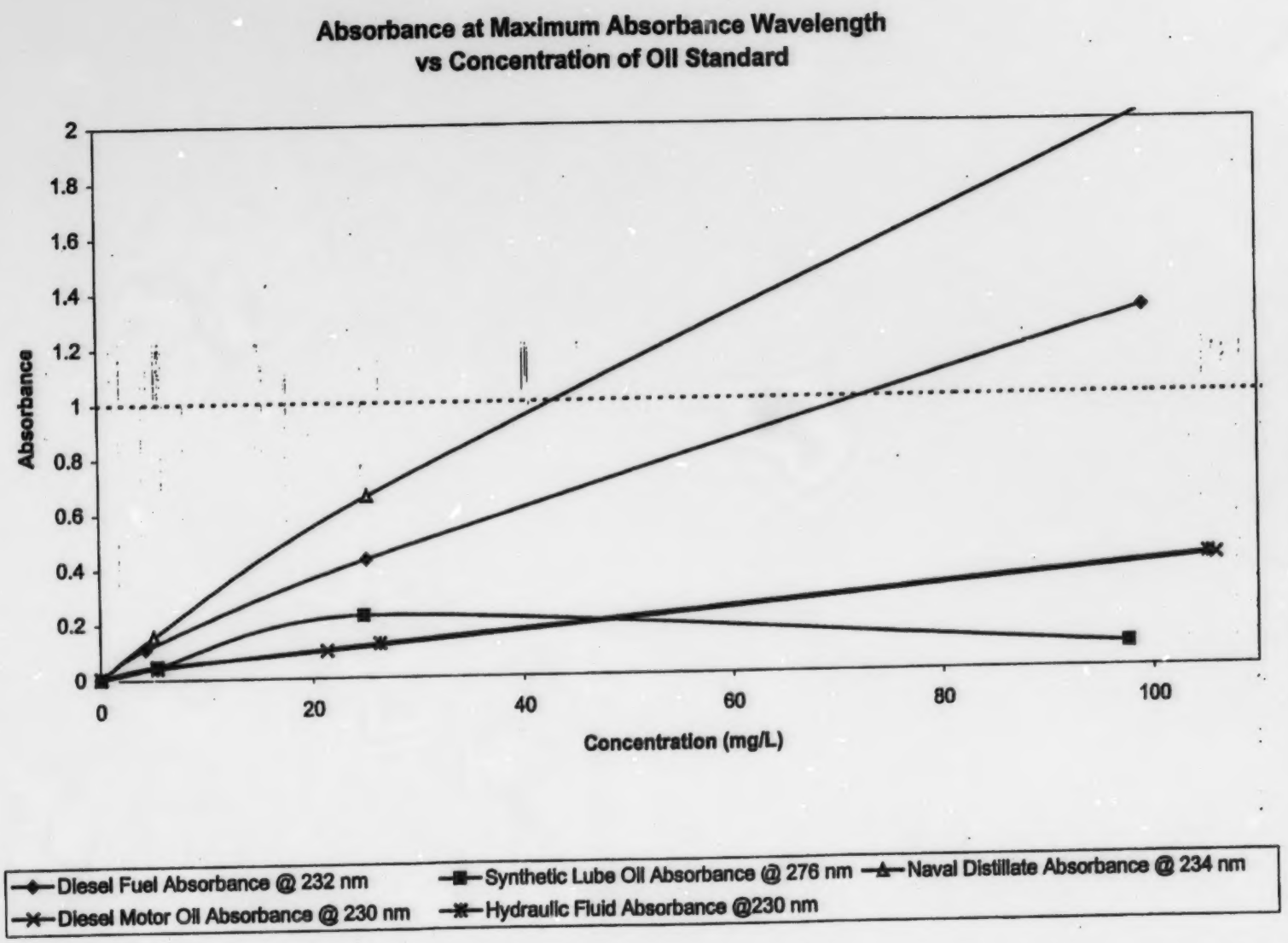
Absorbance at Maximum Absorbance Wavelength
vs Concentration of Oil Standard
Absorbance
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
20
40
60
80
100
Concentration (mg/L)
Diesel Fuel Absorbance @ 232 nm
Synthetic Lube Oil Absorbance @ 276 nm
Naval Distillate Absorbance @ 234 nm
Diesel Motor Oil Absorbance @ 230 nm
Hydraulic Fluid Absorbance @230 nm

Figure 3.2.3

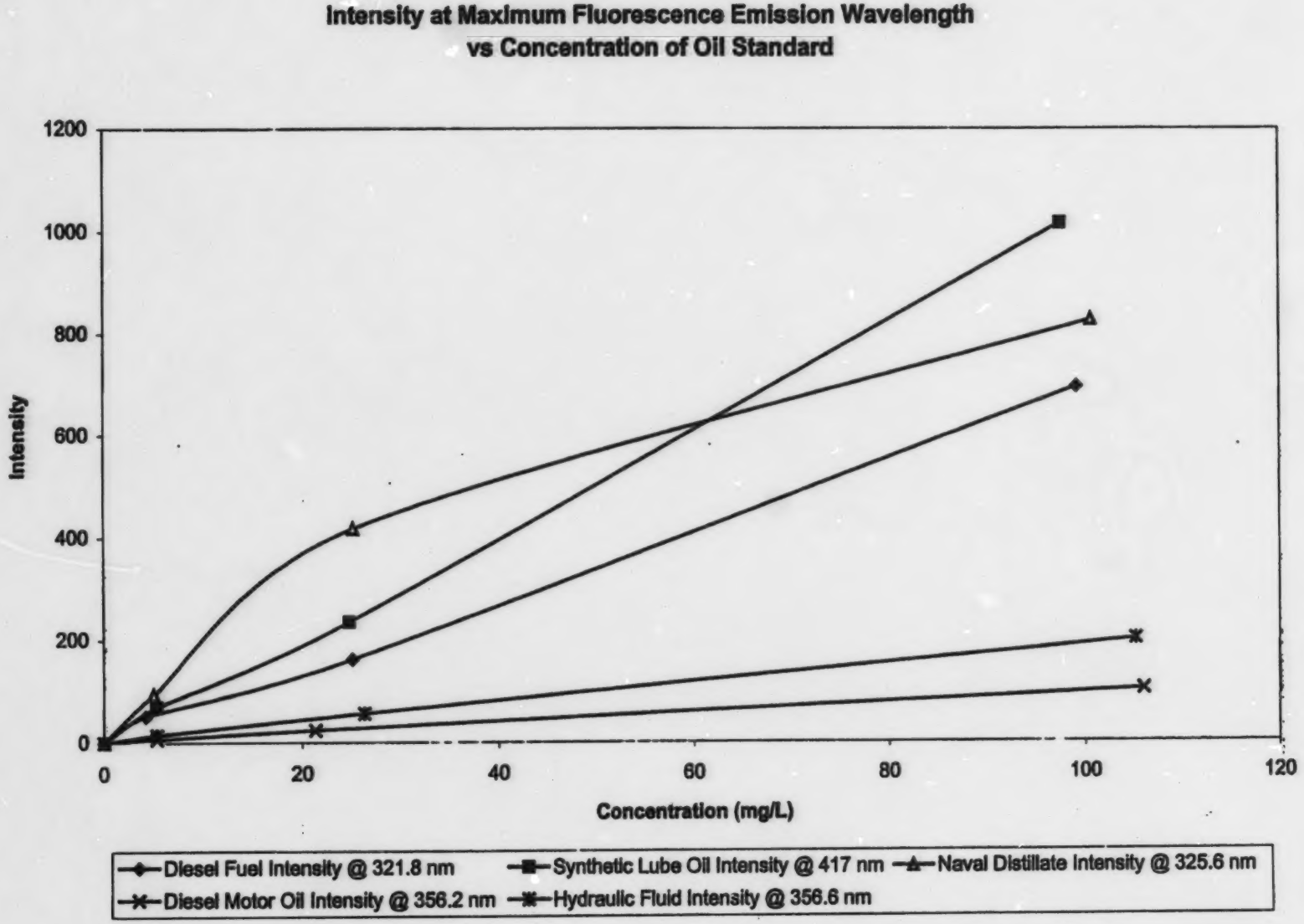
Intensity at Maximum Fluorescence Emission Wavelength
vs Concentration of Oil Standard
Intensity
1200
1000
800
600
400
200
0
0
20
40
60
80
100
120
Concentration (mg/L)
Diesel Fuel Intensity @ 321.8 nm
Synthetic Lube Oil Intensity @ 417 nm
Naval Distillate Intensity @ 325.6 nm
Diesel Motor Oil Intensity @ 356.2 nm
Hydraulic Fluid Intensity @ 356.6 nm

**Figure 3.2.4**

# SEATECH LTD.

*Chemists & Environmental Scientists*

958 Barrington Street, 2nd Floor
Halifax, Nova Scotia, Canada
B3H 2P7
email seatech@seatech.ns.ca

Telephone: (902) 423-5296 Fax: (902) 422-0581
24 HOUR EMERGENCY SERVICE (902) 422-2763


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## Table 3.3.1
## Dissolved Oil in Water Based on U.V. Absorbance

| Sample Identification | Total Oil (mg/L) | Dissolved Oil (mg/L) |
|---|---|---|
| Diesel Fuel | 12.6 | 10.2 |
| Diesel Motor Oil | 13.0 | 2.0 |
| Hydraulic Fluid | 3.3 | 0 |
| Naval Distillate | 5.3 | 0 |
| Synthetic Lube Oil | 54.5 | 17.8 |

Valerie Gray, B.Sc.
Chemist

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# SEATECH LTD.

*Chemists & Environmental Scientists*

958 Barrington Street, 2nd Floor
Halifax, Nova Scotia, Canada
B3H 2P7
email seatech@seatech.ns.ca

Telephone: (902) 423-5296 Fax: (902) 422-0581
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**Table 3.3.2**

## Dissolved Oil In Water Based on U.V. Fluorescence

| Sample Identification | Total Oil (mg/L) | Dissolved Oil (mg/L) |
|---|---|---|
| Diesel Fuel | 27.0 | 20.8 |
| Diesel Motor Oil | 0.2 | 0 |
| Hydraulic Fluid | 11.2 | 6.5 |
| Naval Distillate | 21.4 | 15.1 |
| Synthetic Lube Oil | 17.2 | 14.3 |

Valerie Grey, B.Sc.
Chemist

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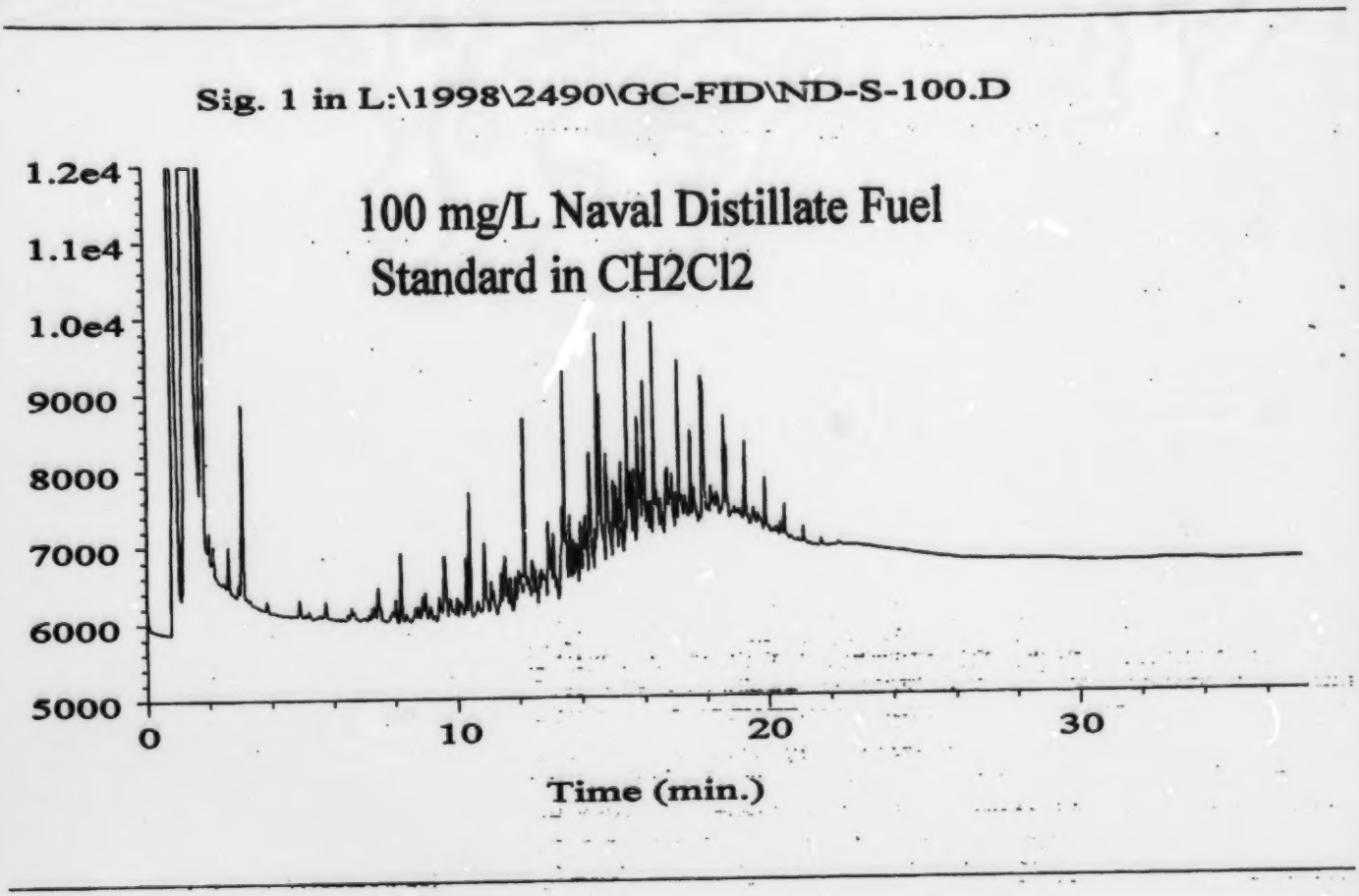

Sig. 1 in L:\1998\2490\GC-FID\ND-S-100.D
100 mg/L Naval Distillate Fuel
Standard in CH2Cl2
1.2e4
1.1e4
1.0e4
9000
8000
7000
6000
5000
0
10
20
30
Time (min.)

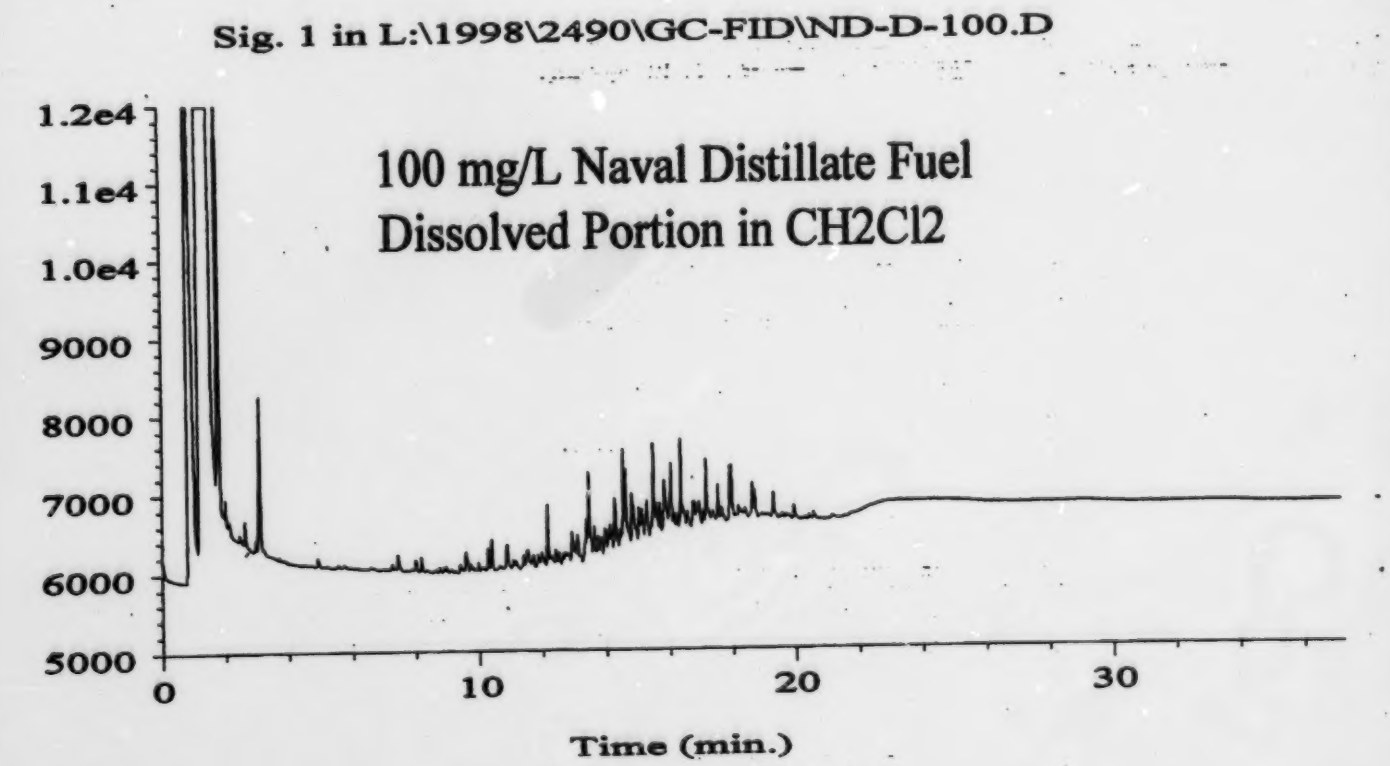
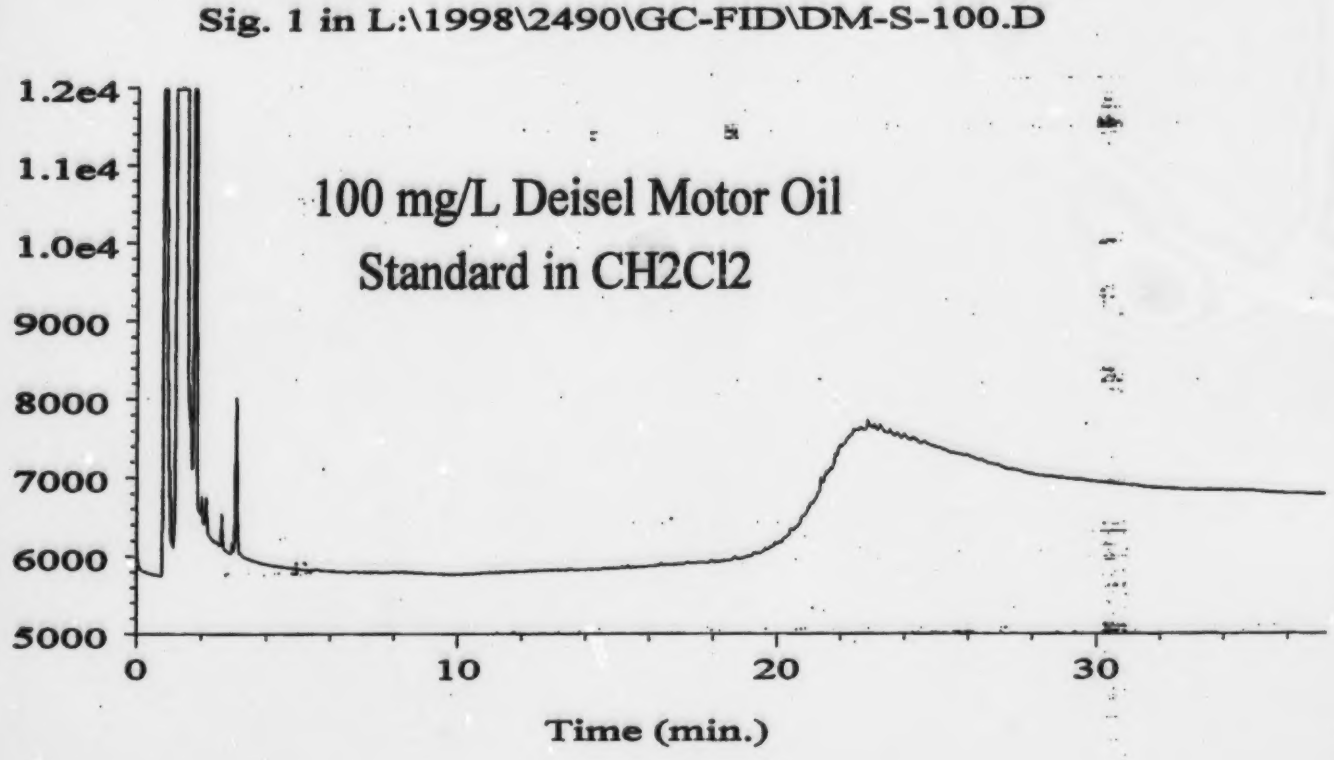

Sig. 1 in L:\1998\2490\GC-FID\ND-D-100.D
100 mg/L Naval Distillate Fuel
Dissolved Portion in CH2Cl2
1.2e4
1.1e4
1.0e4
9000
8000
7000
6000
5000
0
10
20
30
Time (min.)

Figure 3.4.1


Sig. 1 in L:\1998\2490\GC-FID\DM-S-100.D
1.2e4
1.1e4
1.0e4
9000
8000
7000
6000
5000
100 mg/L Deisel Motor Oil
Standard in CH2Cl2
0
10
20
30
Time (min.)

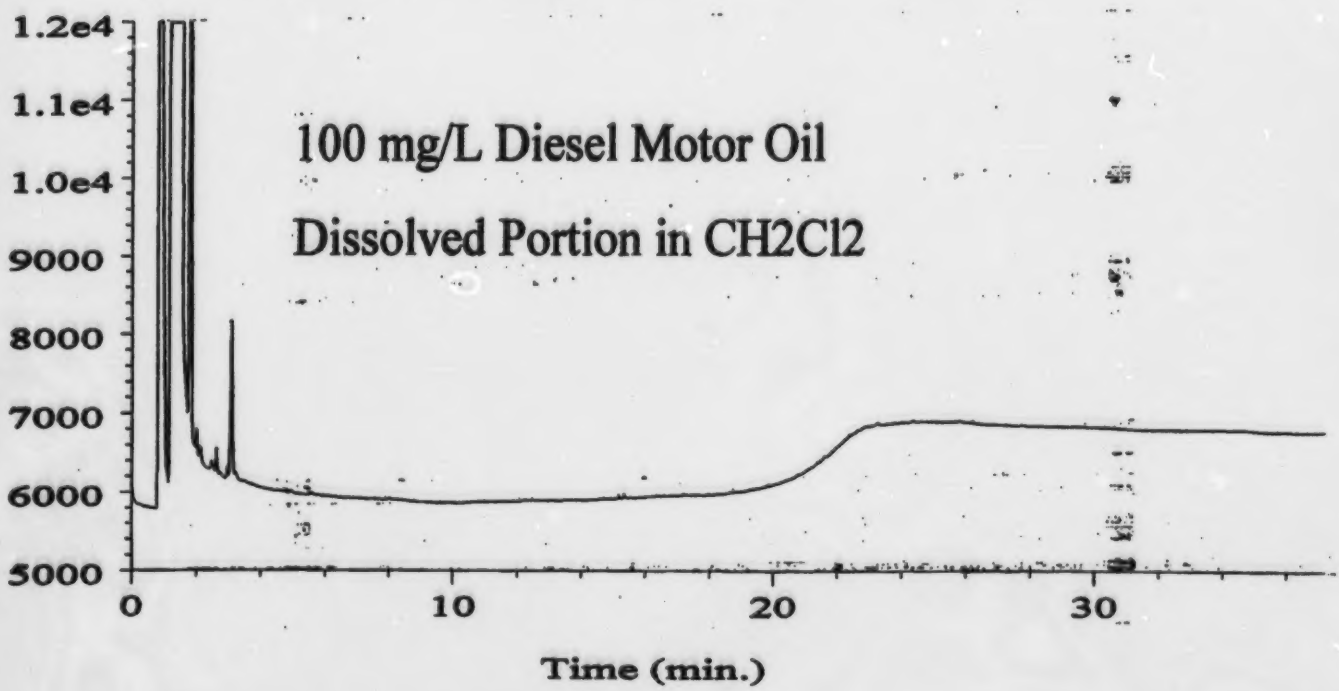

Sig. 1 in L:\1998\2490\GC-FID\DM-D-100.D
1.2e4
1.1e4
1.0e4
9000
8000
7000
6000
5000
100 mg/L Diesel Motor Oil
Dissolved Portion in CH2Cl2
0
10
20
30
Time (min.)

**Figure 3.4.2**

after one, six and twenty-four hour time intervals. Sample concentrations, based on UV absorbance at 235 nm, of the different time intervals were then compared (Table 3.5.1). It was found that maximum dissolution had been obtained in one hour. In the case of the fuel oils the dissolved portions remained unchanged after one hour, when allowing for sample variability. In the case of the heavier petroleum products (lubricating and hydraulic oils) the amount of dissolved oil appeared to decrease over time. This was believed to occur due to the adherence of oil to the glass walls of the mason jar.

### 3.5 Accuracy, Minimum Detection Limits and Linearity of Response

Figures 3.6.1 to 3.6.5 show absorbance and fluorescence response for the oil standards in methylene chloride, as measured at the selected fixed wavelengths. Measurements at these same wavelengths for oil in water as total portion (particulate and dissolved), and dissolved portion are also presented in these figures. In a very accurate measurement the total as measured directly in water should be the same for the standards in methylene chloride.

For Diesel Fuel (Figure 3.6.1) at less than 25 mg/L UV absorbance tended to under report by up to 40%, while UV fluorescence over reported oil in water content slightly. For Naval Distillate (Figure 3.6.2) at less than 25 mg/L UV absorbance under reported by 60%, while UV fluorescence under reported by only 10 to 20%. For Diesel Motor Oil (Figure 3.6.3) at less than 25 mg/L UV absorbance under reported by 50%, while UV fluorescence under reported by over 75%. For Hydraulic Fluid (Figure 3.6.4) at less than 25 mg/L UV absorbance under reported by up to 75%, while UV fluorescence under reported by 50%. For Synthetic Lube Oil (Figure 3.6.5) at less than 25 mg/L. UV absorbance varied widely and over reported by up to 300%, while UV fluorescence under reported by up to 25%.

A calculated minimum detection limit can be determined by taking the noise of the blank (in UV absorbance units or UV fluorescence intensity units) as a standard deviation and doubling (ie the 2N level). Precision was estimated based on one standard deviation at the blank level of oil in water. This absorbance or fluorescence intensity was then converted to mg/L of oil in water at the two wavelengths (UV absorbance = 235 nm UV fluorescence emissions = 380 nm). The results are noted below (based on n=7):

Minimum detection limit, UV absorbance = 1.4 mg/L, precision = ±0.7 mg/L
Minimum detection limit, UV fluorescence = 0.3 mg/L, precision = ±0.13 mg/L

# SEATECH LTD.

*Chemists & Environmental Scientists*

958 Barrington Street, 2nd Floor
Halifax, Nova Scotia, Canada
B3H 2P7
email seatech@seatech.ns.ca

Telephone: (902) 423-5296 Fax: (902) 422-0581
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**Table 3.5.1**

## Time Trials To Determine Maximum Dissolution of Oil in Seawater

| Total Oil Concentration | Time Shaken | Concentration of Dissolved Oil (mg/L)* |
|---|---|---|
| 20 ppm Diesel Fuel | 1 hr | 10.0 |
| | 6 hr | 9.3 |
| | 24 hr | 10.4 |
| 20 ppm Diesel Motor Oil | 1 hr | 3.2 |
| | 6 hr | 1.4 |
| | 24 hr | 0.2 |
| 20 ppm Hydraulic Fluid | 1 hr | 1.6 |
| | 6 hr | 0.5 |
| | 24 hr | 0.0 |
| 20 ppm Naval Distillate Fuel | 1 hr | 4.0 |
| | 6 hr | 2.0 |
| | 24 hr | 6.0 |
| 20 ppm Synthetic Lube Oil | 1 hr | 1.1 |
| | 6 hr | 0.9 |
| | 24 hr | 1.1 |

* Based on U.V. absorbance at 235 nm.

***Average concentration at 1 hour = 3.9 ppm***
***Average concentration at 6 hour = 2.8 ppm***
***Average concentration at 24 hour = 3.4 ppm***

Based on these averages, a one hour shaking time was chosen.

Valerie Gray, B.Sc.
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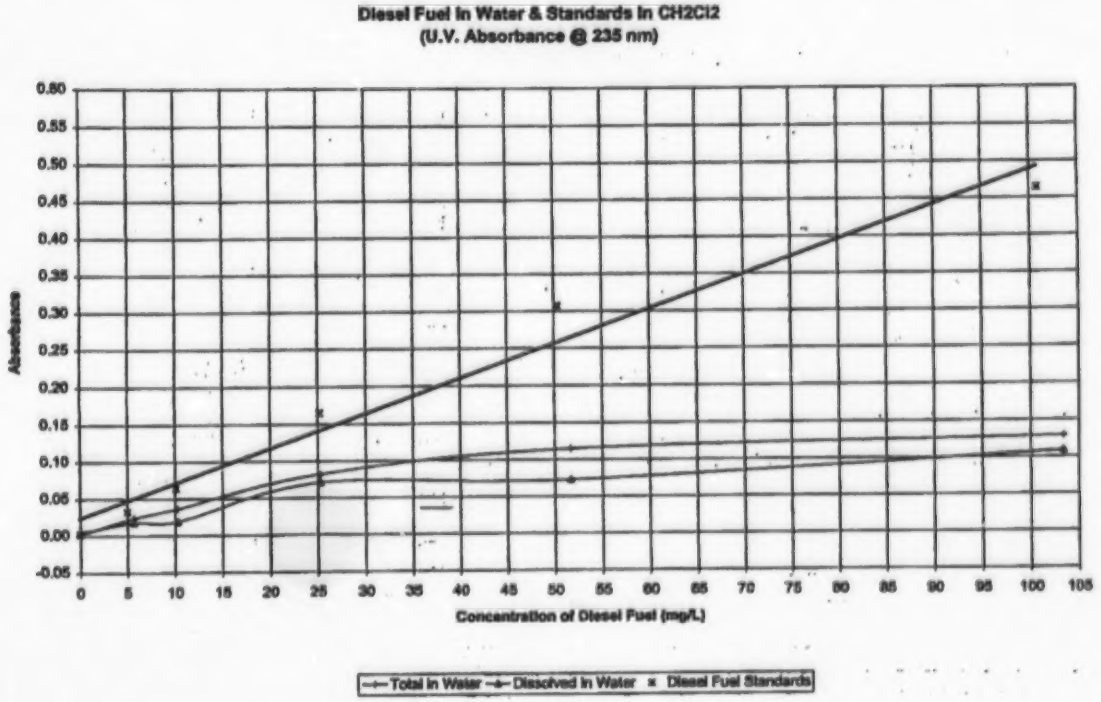

Diesel Fuel in Water & Standards in CH2Cl2
(U.V. Absorbance @ 235 nm)
Absorbance
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-0.05
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105
Concentration of Diesel Fuel (mg/L)
Total in Water
Dissolved in Water
Diesel Fuel Standards

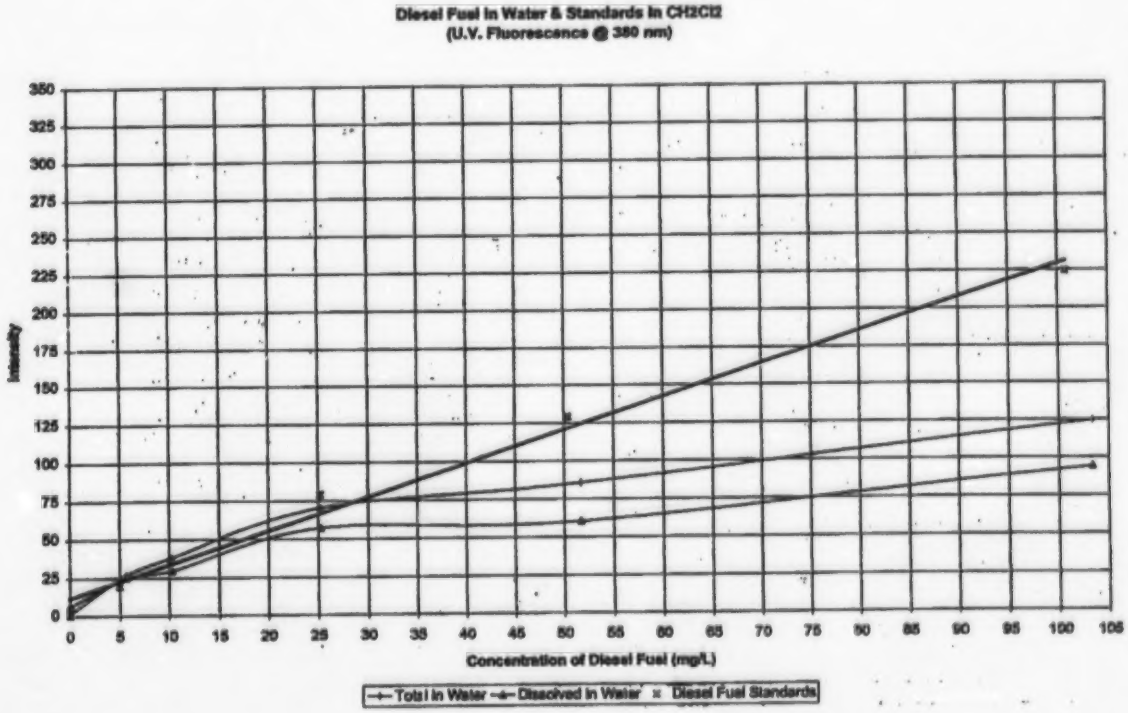
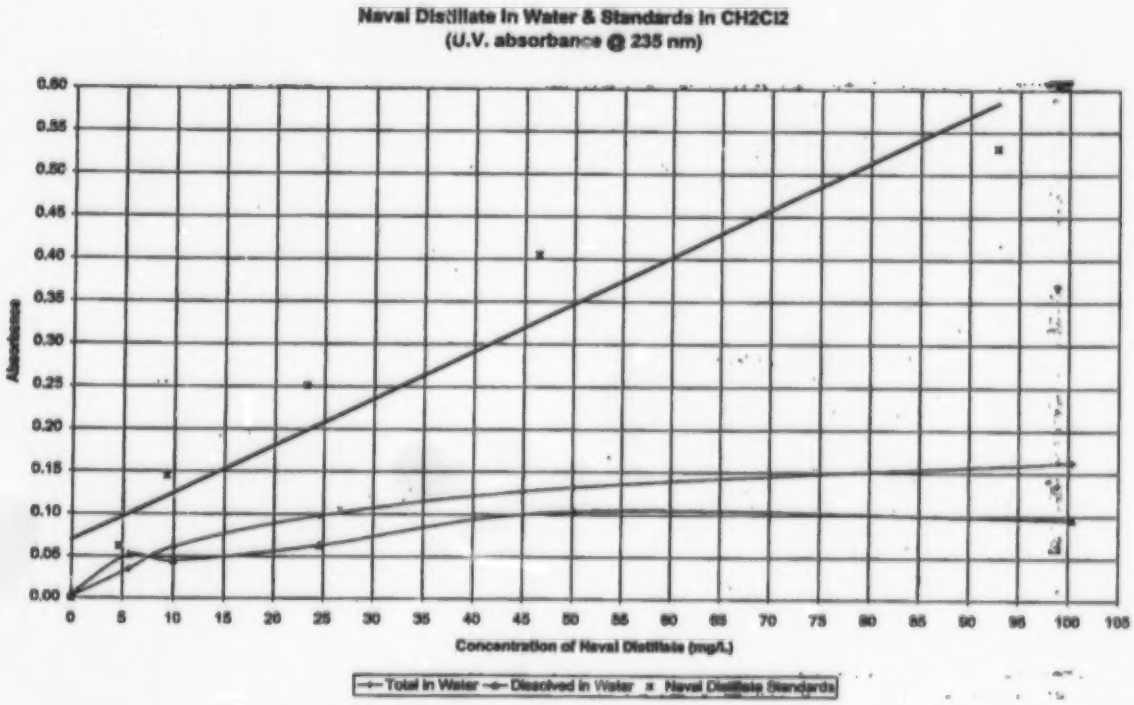

Diesel Fuel in Water & Standards in CH2Cl2
(U.V. Fluorescence @ 380 nm)
Intensity
350
325
300
275
250
225
200
175
150
125
100
75
50
25
0
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105
Concentration of Diesel Fuel (mg/L)
Total in Water
Dissolved in Water
Diesel Fuel Standards

Figure 3.6.1


Naval Distillate in Water & Standards in CH2Cl2
(U.V. absorbance @ 235 nm)
Absorbance
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105
Concentration of Naval Distillate (mg/L)
Total in Water
Dissolved in Water
Naval Distillate Standards

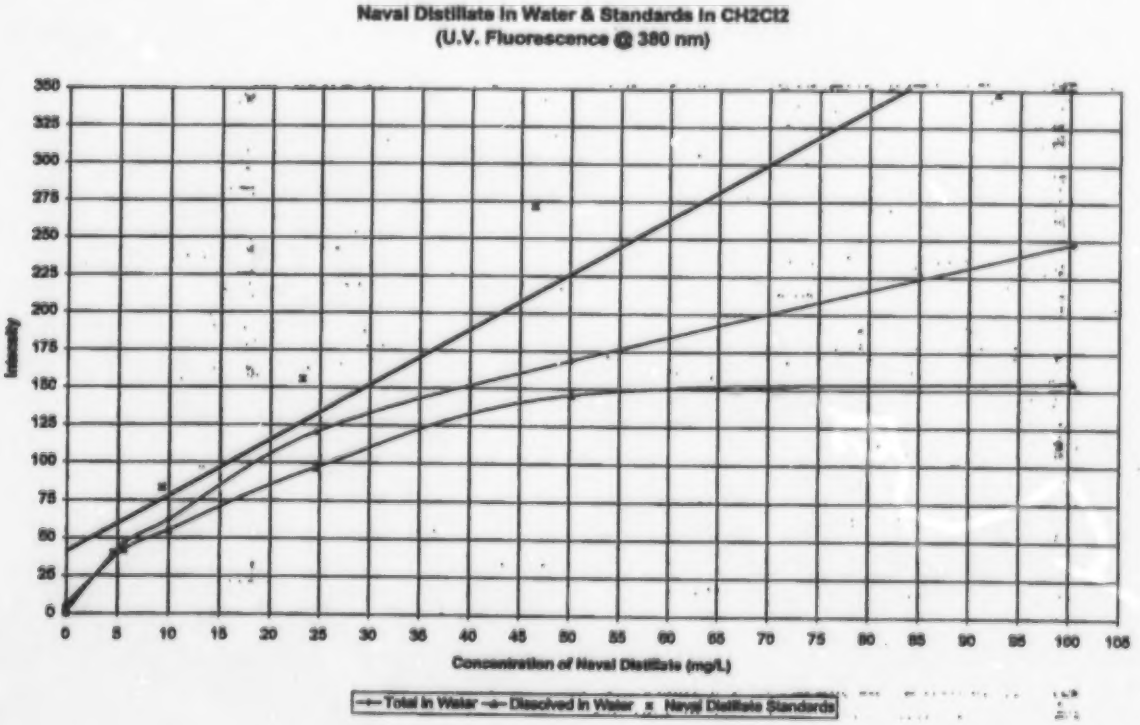
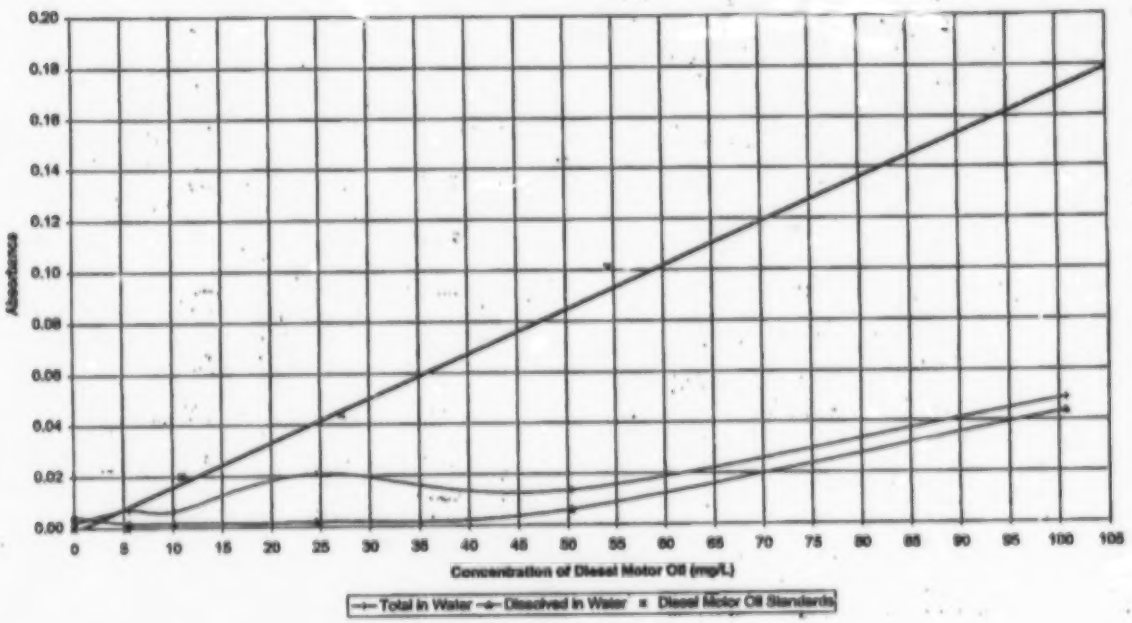

Naval Distillate in Water & Standards in CH2Cl2
(U.V. Fluorescence @ 380 nm)
Intensity
350
325
300
275
250
225
200
175
150
125
100
75
50
25
0
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105
Concentration of Naval Distillate (mg/L)
Total in Water
Dissolved in Water
Naval Distillate Standards

Figure 3.8.2

**Diesel Motor Oil in Water & Standards in CH2Cl2**
**(U.V. Absorbance @ 235 nm)**




**Diesel Motor Oil in Water & Standards in CH2Cl2**
**(U.V. Fluorescence @ 380 nm)**

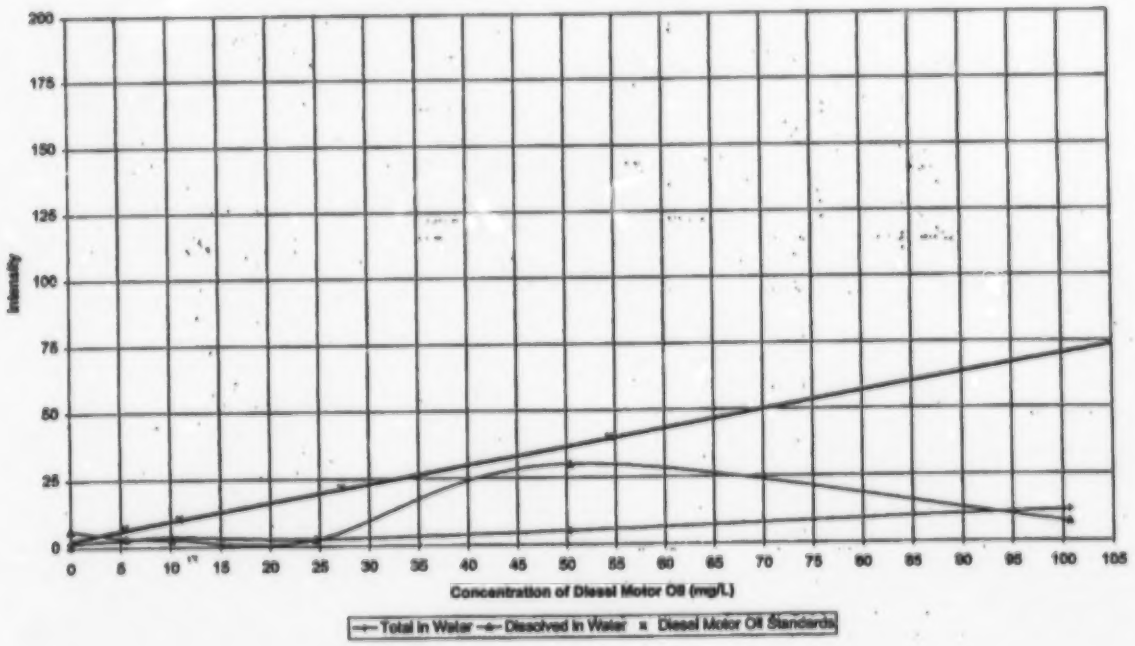
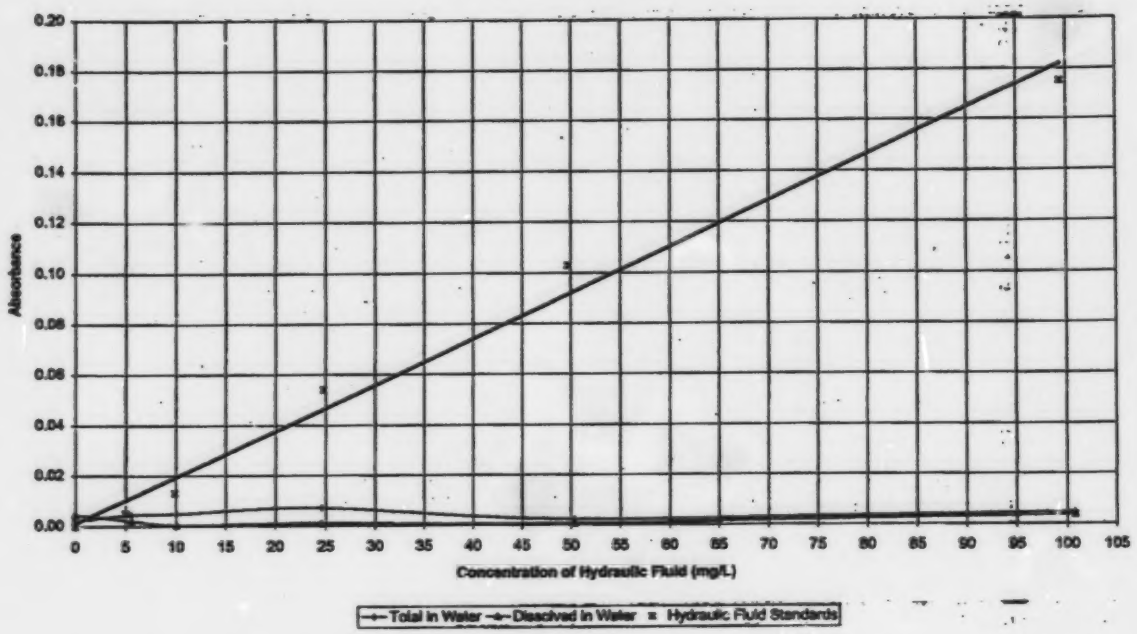



**Figure 3.6.3**

**Hydraulic Fluid in Water & Standards in CH2Cl2**
**(U.V. Absorbance @ 235 nm)**




**Hydraulic Fluid in Water & Standards in CH2Cl2**
**(U.V. Fluorescence @ 380 nm)**

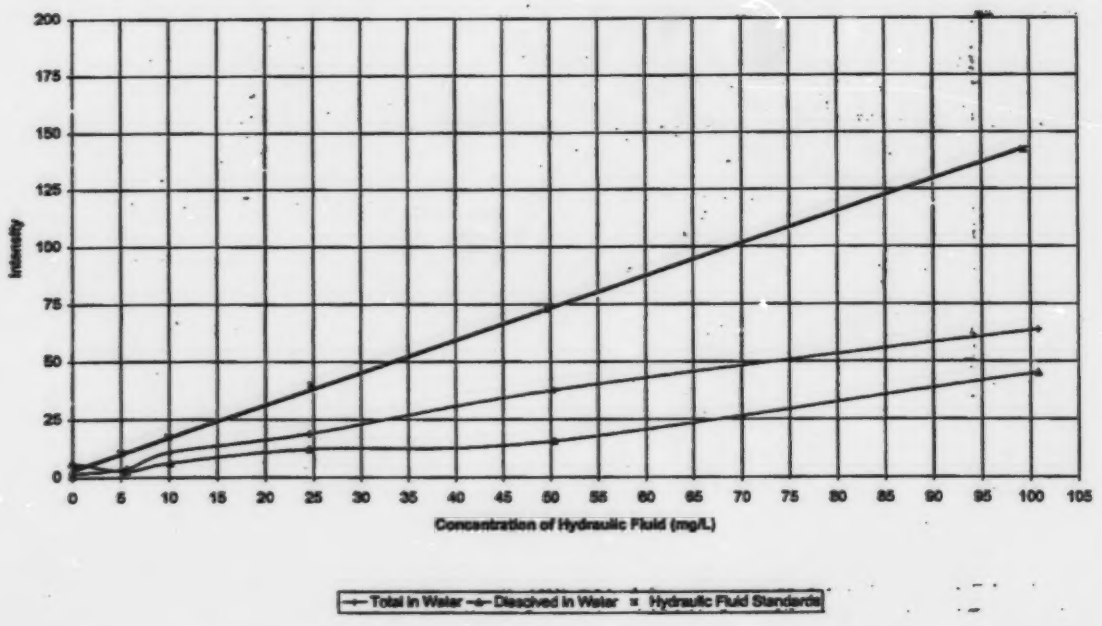
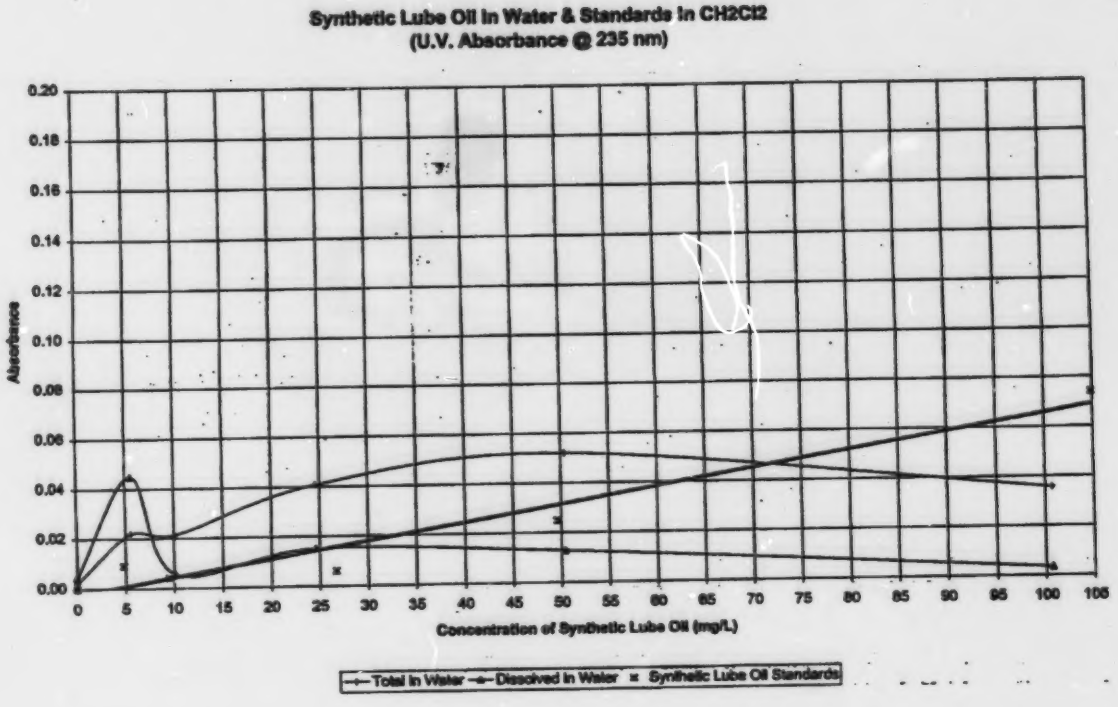



**Figure 3.6.4**


Synthetic Lube Oil in Water & Standards in CH2Cl2
(U.V. Absorbance @ 235 nm)
Absorbance
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105
Concentration of Synthetic Lube Oil (mg/L)
Total in Water
Dissolved in Water
Synthetic Lube Oil Standards

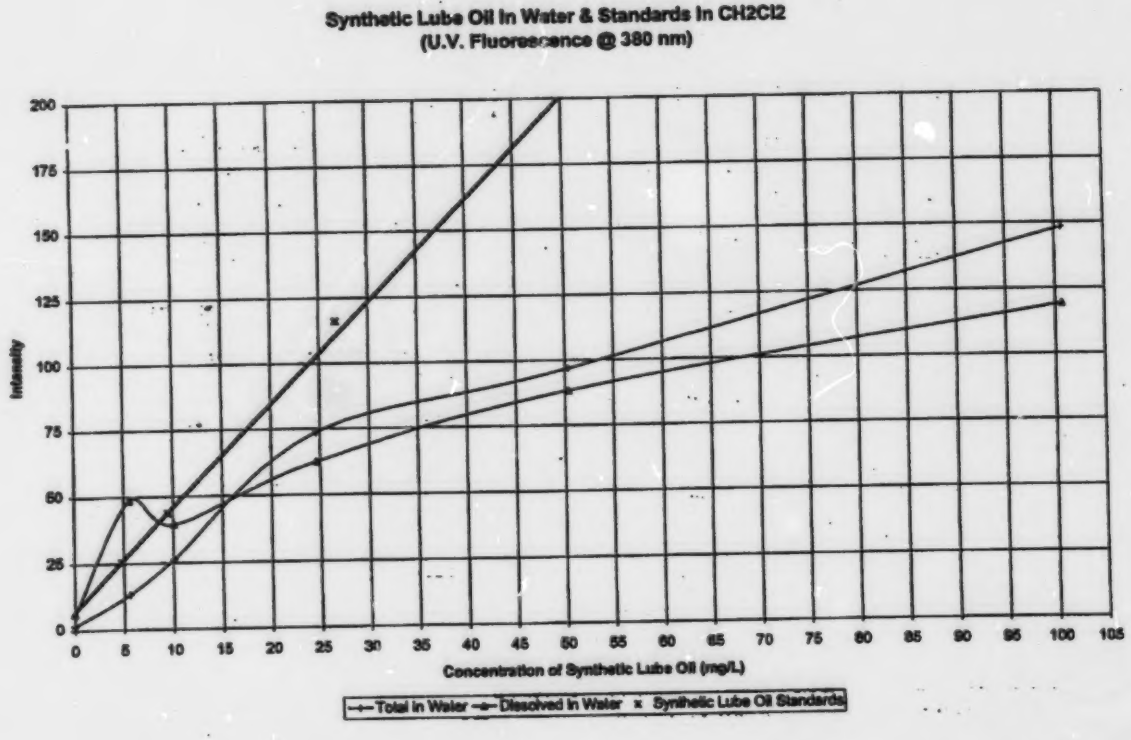

Synthetic Lube Oil in Water & Standards in CH2Cl2
(U.V. Fluorescence @ 380 nm)
Intensity
200
175
150
125
100
75
50
25
0
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105
Concentration of Synthetic Lube Oil (mg/L)
Total in Water
Dissolved in Water
Synthetic Lube Oil Standards

Figure 3.6.5

These results were for a static reading in a cuvett (ie not flow through).

Linearity of response with concentration was determined by the correlation coefficient ($R^2$) for the least squares, straight line fit for each oil as measured by UV absorbance or fluorescence versus concentration of oil in water. (From Figures 3.6.1 - 3.6.5) Table 3.6.1 presents this information for the five oil types. UV fluorescence always had a much better least squares fit then UV absorbance for all oils in water.

### 3.6 Testing Of Shipboard Samples

The five bilge and tank water bottom samples primarily contained Naval Distillate Fuel and Lube Oils. Oil in water concentrations were determined by GC-FID of Naval Distillate varied from 7 mg/L in the IAC Plant to 3.5 % in the #5 Settling Tank sample (Table 3.7.1).

These waters were tested for oil in water by UV absorbance and UV fluorescence, measurements made directly on the samples in a cuvett. Results are presented in Table 3.7.2. Concentrations of total oil in water were calculated based on two different standard curves used in Figures 3.6.2 and 3.6.3 (Naval Distillate and Diesel Motor Oil). Except for the IAC Plant Bilge, all oils in water as determined in the lab were much greater then 100 mg/L (upper end of calibration range). Not surprisingly these high levels outside the calibration range were significantly under reported by both UV absorbance and UV fluorescence. The IAC Plant Bilge at 7 mg/L lube oil was closest estimated by UV fluorescence (49 mg/L), but still with large discrepancy in over reporting actual concentration of oil present.

SEATECH LTD.
*Chemists & Environmental Scientists*
958 Barrington Street, 2nd Floor
Halifax, Nova Scotia, Canada
B3H 2P7
email seatech@seatech.ns.ca
Telephone: (902) 423-5296 Fax: (902) 422-0581
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**Table 3.6.1**

## Regression Coefficients of Least Squares for U.V. Absorbance and Fluorescence of Oils in Water

| Oil Type | U.V. Absorbance $R^2$ (Absorbance @ 235 nm) | U.V. Fluorescence $R^2$ (emission @ 380 nm) |
|---|---|---|
| Diesel Fuel | 0.81 | 0.90 |
| Naval Distillate Fuel | 0.84 | 0.94 |
| Diesel Motor Oil | 0.88 | 0.92 |
| Hydraulic Fluid | 0.01* | 0.99 |
| Synthetic Lube Oil | 0.36* | 0.93 |

$R^2$ calculated over the range 0 to 100 mg/L of each oil.

* Low $R^2$ are primarily caused by the flat (non-response) nature of the absorbance versus concentration curve (see Figures 3.6.4 and 3.6.5)

Valerie Gray, B.Sc.
Chemist

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# SEATECH LTD.

*Chemists & Environmental Scientists*

958 Barrington Street, 2nd Floor
Halifax, Nova Scotia, Canada
B3H 2P7
email seatech@seatech.ns.ca

Telephone: (902) 423-5296 Fax: (902) 422-0581
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**Table 3.7.1**

## OIL IN SHIPBOARD WATER SAMPLES (HMCS ATHABASCAN, MARCH 1998)

| Location | Total Petroleum Hydrocarbon* (mg/L) | Dissolved Petroleum Hydrocarbon * (mg/L) | TYPE OF OIL |
|---|---|---|---|
| AMR Bilge | 844 | 53 | Primarily Naval Distillate |
| IAC Plant Bilge | 7.3 | 4.7 | Primarily Lube Oil |
| #5 Settling Tank | 25,800 | 35,500[1] | All Naval Distillate |
| Aft Stripping Tank | 680 | 109 | Naval Distillate |
| Gland Space Bilge | 3,880 | 148 | Primarily Lube Oil |
| Composite | 12,500 | 6,334 | Primarily Naval Distillate |

Samples collected by Seatech

* Dissolved petroleum hydrocarbons determined on subsurface subsample taken from sample after standing one hour, to permit particulate oil to float to the top of the sample container. TPH Determined by cold solvent extraction and GC-FID.

1. Suspect subsurface sample (15 mL) contaminated by large amount of free floating surface oil collecting on pipette used for subsurface sample.

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Table 3.7.2

## TOTAL OIL IN SHIPBOARD WATER SAMPLES AS DETERMINED BY U.V. ABSORBANCE AND FLUORESCENCE (HMCS ATHABASCAN, MARCH 1998)

| Location | Total Oil by GC-FID (mg/L) | U.V. Absorbance Calculated as Naval Distillate (mg/L) | U.V. Absorbance Calculated as Diesel Motor Oil (mg/L) | U.V. Fluorescence Calculated as Naval Distillate (mg/L) | U.V. Fluorescence Calculated as Diesel Motor Oil (mg/L) |
|---|---|---|---|---|---|
| AMR Bilge | 844 | 72 | 274 | 71 | 443 |
| IAC Plant Bilge | 7.3 | 54 | 217 | 0 | 49 |
| #5 Settling Tank | 25,800 | 113 | 410 | 41 | 281 |
| Aft Stripping Tank | 680 | 101 | 369 | 104 | 622 |
| Gland Space Bilge | 3,880 | 95 | 348 | 77 | 478 |
| Composite | 12,500 | 107 | 389 | 118 | 699 |

Samples collected by Seatech

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Chemist

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## 4.0 Discussion and Conclusions

### 4.1 Literature

There are three types of detectors in common use for oil detection monitors described in the literature. White light and infrared detectors were the historical monitors of choice. However, only UV detectors had the capability to detect dissolved oil at levels below 5-10 ppm.

The most common UV spectroscopic technique was found to be fluorescence. Levels of detection for fluorescence were reported at 1 ppm and less, which is sufficient for the expected Canadian Naval requirement.

### 4.2 UV Absorbance and Fluorescence Spectra

The UV absorbance spectra showed maximum light absorbance for fuel oils (Diesel and Naval Distillate) at 232-234 nm, while Synthetic Lube showed a maxima at 276 nm (Figure 3.2.1) Both Diesel Motor Oil and Hydraulic Fluid exhibited only weak absorption maxima anywhere in the UV spectrum (190-390 nm), and were strongest at 230 nm, but with a relative absorbance intensity of only 1/5 to 1/8 that of the fuel oils. Thus there was no common UV absorption maxima wavelength that could apply to all oils expected in Naval water discharges. At the compromise wavelength chosen for oil quantification by UV absorbance (235 nm), the response varied by a factor of 16 from Hydraulic Fluid (lowest response) to Naval Distillate (highest response). The Diesel Motor Oil (lubes) and Hydraulic Fluid showed the least response with concentration (see Figure 3.2.3). This lack of response with varying concentration of oil in water was to such an extent as to make UV absorbance unacceptable for purposes of quantification of Diesel Motor Oil or Hydraulic Fluid in discharge water.

The UV fluorescence emission spectra (excitation 254 nm) showed maximum emission at 320-325 nm for fuel oils (Diesel and Naval Distillate), while synthetic lube showed a maximum emission at 417 nm. Both Diesel Motor Oil and Hydraulic Fluid exhibited only weak emission maxima at 356 nm (Figure 3.2.2) Thus, for fluorescence also, there was no common emission maximum wavelength that could apply to all oils expected in Naval water discharges. A compromise emission wavelength of 380 nm was chosen for oil quantification. At 380 nm emission wavelength chosen as a compromise response varied by a factor of 10 from Diesel Motor Oil (lowest response) to Naval Distillate and Synthetic Lube (highest response) . Diesel

Motor Oil and Hydraulic Fluid showed a very low response with concentration (Figure 3.2.4), but not as poor as that for UV absorbance.

In conclusion there is no single wavelength or even a broad band wavelength (say ± 20 nm) that would accurately quantify all oils. At best fuel oils can be reasonably quantified at one wavelength and lubes/hydraulics at another wavelength. Fluorescence gave a better response to varying concentration for all oils compared to UV absorbance.

### 4.3 Maximum Dissolution of Oil

Based on UV absorbance measurements of oil at 25 mg/L or less the percent dissolved averaged 18% with a range from 0 to 81%. Based on UV fluorescence the percent dissolved averaged 74% with a range from 0 to 83%. Concurrent lab analyses at 100 mg/L showed an average 30% dissolved. In general lower levels of oil had higher percent dissolved and fuel oils (Naval Distillate and Diesel) showed higher percent dissolved than Lube or Hydraulic Fluids. These findings were consistent with the literature found.

The "real" samples having total oil concentrations of several hundred to several thousand mg/L and had an average percent dissolved of 51% (ranging from 4% to 100%). The only sample with less than 100 mg/L contained Lube Oil at 7.3 mg/L of which 64% was dissolved.

The rate of dissolution was found to be quite rapid, being one hour, based on shaking tests. Longer shaking did not dissolved more oil in water phase. To detect all oil in discharge waters it is imperative to be able to detect dissolved oil at less than 10 mg/L.

### 4.4 Accuracy, Minimum Detection Limits and Linearity

The accuracy of both UV absorbance and fluorescence varied significantly with oil type and concentration based on use of a single wavelength for all oils. The biggest obstacle to accuracy is the varying type of oils or mixture of types in the expected discharges. Considering that different oils have different absorbance and fluoresence maxima an acceptable accuracy would be ±20% due to changing response with concentration. In fact UV absorbance (at <25 mg/L) either under reported by 40-60% or over reported by 300%. For UV fluorescence the situation was only slightly better, under reporting by 10-50% in most cases. The problem however, is not the UV spectroscopy but the choice of calibration oil (or oil mixture) and

calibration wavelength. The UV spectroscopy technologies work best where a uniform composition oil is being discharged (eg oil production rig on a specific oil field).

The desired minimum detection limit of 1 ppm was achieved easily by UV fluorescence but was marginally exceeded by UV absorbance. The linearity of response over 0-100 mg/L was considerably better for UV fluorescence, compared to UV absorbance.

### 4.5 Summary

The study had two main objectives. The first objective of the study was to undertake spectral scans of various typical Naval oils in several states (extracted, total in water, dissolved in water) to determine whether this spectral range would be useful for oil detection and what would be the limits of detection. The answer to this objective was that both UV fluorescence and UV absorbance will work as detectors for oil in water at levels below 10 ppm. Fluorescence is the detector of choice for three reasons:

1) Better linearity of response for the various oil types.
2) A better detection limit.
3) Will not respond to detergents, whereas absorbance detectors may misinterpret these additives as oil and give a false high oil in water concentration. (based on literature)

The second objective was to determine the dissolved portion of these Naval oils and the ability of this technology to accurately measure these levels in water. It has been demonstrated in this project that there is a dissolved fraction of oil in the water phase varying on average from 25-75% depending on oil type (low dissolution for lubes, higher dissolution for distillates) . Instruments which employ light scattering techniques would be ineffective in measuring the low levels of dissolved oil. UV fluorescence would be the technique most capable of this type of measurement. Accuracy for all oils was generally outside the limits of ±20% for both detection options (absorbance and fluorescence). This was due entirely to lack of an appropriate calibration standard and choice of an appropriate wavelength or wavelengths for measurement.

**In conclusion, the UV fluorescence technique is preferred over the other techniques for direct monitoring of oil in water. It is confirmed that a large portion of any discharged oil will be in dissolved form. Calibration is the single largest problem in producing an accurate UV spectroscopic based oil detection monitor.**

## 5.0 Recommendations

1. UV Fluorometry is the method of choice, though UV absorbance can not be discounted. Fluorometry was better for level of detection and linearity at low oil in water levels. Testing of flow through fluorometry on samples typical of Canadian Naval requirements is recommended since the oil water mixtures are somewhat complex (ie different). This testing should target choice of calibration wavelengths and standard oil mix. Getting an Oil Detection Monitor to provide an accurate value (even ±10 ppm) is questionable, due to varying input oil type. We would recommend a more detailed assessment of oil types and concentrations expected to be delivered to this unit. In addition, during this work, flow through oil in water fluorometry on real samples as might be expected from the treatment unit should be undertaken to confirm response of the unit in a generic sense. We understand a Sigrist flow through oil in water fluorometer would also be available for testing, and we would recommend carrying out this testing on that unit as well as a laboratory unit. The purpose of this testing would be to determine a scientifically supportable calibration mixture, wavelength and procedure.

2. There are off the shelf fluorometer monitors (Turner & Sigrist) with unit costs of $15,000-20,000 US dollars. It is estimated a Canadian fluorometer could be developed from an existing technology base (ie experience in construction of absorbance spectrophotometer) for $125,000-150,000 of development funding. The units are anticipated to sell for $11,000-12,000 Canadian. On development and purchase of 20 units this would be a total cost of $390,000 Canadian, versus the purchase option of $420,000 Canadian, based on the higher priced US units (Turner or Sigrist). Consideration should be given to this option for development of a unit in two stages. The first stage would be an fluorometry operating prototype for $75,000, then an amended prototype to DND specifications for fit to the oil in water separator ($50,000-75,000).

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Defence Research Establishment Atlantic contracted Seatech Ltd. To assess the capability of UV spectrophotometry to detect oil in water as may be discharged from DND vessels.

For the study a brief literature review was undertaken and five oils (Diesel Fuel, Naval Distillate, Diesel Motor Oil, Synthetic Lubricating Oil, Hydraulic Fluid) assessed over the range 5 to 100 ppm in water. In addition some "real" samples from a naval vessel at Halifax were tested by UV spectrophotometry and standard methods for oil content.
The findings were as follows:

1. Both UV absorbance and fluorescence will detect oil in water in dissolved and particulate phases at concentrations below 10 ppm, with a detection limit of approximately 1 ppm.
2. The portion of oil dissolved, compared to the total oil in water varied from 25% to 75%, with greater dissolution for fuel oils and lesser for lubes/hydraulics.
3. Wavelength of maximum UV absorbance or fluorescence, and response per unit concentration varied with oil type: thus making the choice of a standard for calibration of an oil detection monitor difficult.

It was determined that response versus oil in water concentration, linearity, and minimum level of detection were better for UV fluorescence than for UV absorbance. The literature review indicated UV fluorescence was generally preferred over UV absorbance and other detectors, and was less subject to interferences from bilge chemicals or solids compared to UV absorbance. "Real" samples were found to contain very high levels of oil in water and UV spectrometry did not accurately estimate these levels of oil. UV spectrophotometry, particularly UV fluorescence, was most accurate at 25 ppm or less oil in water.

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